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Indian Standard

SPECIFICATION FOR ZINC PHOSPHATE DENTAL CEMENT

(First Revision)

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Indian Standard

SPECIFICATION FOR ZINC PHOSPHATE DENTAL CEMENT

(First Revision)

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Indian Standard

SPECIFICATION FOR ZINC PHOSPHATE DENTAL CEMENT

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 28 February 1986, after the draft finalized by the Dental Material Sectional Committee had been approved by the Chemical Division Council.

0.2 The phosphate dental cements are used as luting agents to seal dental appliances to hard oral structures or to other appliances. These can also be used as a base for tooth-filling material and as a temporary filling material by increasing the ratio of powder to liquid relative to that used for luting.

0.3 Dental zinc phosphate cements are based upon the reaction between an oxide powder, the principal constituent of which is zinc oxide and an aqueous solution of phosphoric acid, which may contain metal ions.

0.4 This standard was first published in 1970 and was based upon ISO/R 1566, prepared by International Organization for Standardization (ISO). The ISO recommendation has now been revised as ISO 1566-1978 'Dental zinc phosphate cement' and hence this revision.

0.4.1 Of the various changes introduced in this revision, most stem from the use of a much smaller test specimen than used previously. The reason for using the small specimen is to align the test methods for hand-mixed materials as closely as possible with those for capsulated materials. In general this has merely necessitated some adjustments in technique to accommodate the smaller test specimen, but in some instances, the water-leachable material test in particular, more basic changes in the test method have also been made.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for hand-mixed zinc phosphate cement.

1.1.1 The primary uses of this material are:

a) to join or to seal dental appliances to hard oral structures or to other appliances,

b) to serve as a base for a tooth filling material, and

c) to serve as a temporary filling material by increasing the ratio of powder to liquid relative to that used for luting.

2. TYPES

2.1 Zinc phosphate cement shall be of the following two types:

a) Type 1 — Fine grain, and

b) Type 2 — Medium grain.

2.1.1 Each type shall further be of the following classes:

a) Class 1 — Fast setting, and

b) Class 2 — Normal setting.

3. REQUIREMENTS

3.1 Description — The cement shall consist of a powder and a liquid which, when mixed according to the manufacturer's instructions, will set rapidly to a condition suitable for its intended use.

3.2 Liquid — The liquid shall be free from obvious deposits or filaments on the inside of its container.

3.3 Powder — The powder shall be free from extraneous material and if the powder is coloured, the pigment shall be uniformly dispersed throughout the powder.

3.4 Unset Cement — The cement when mixed as directed in **A-1.3** shall be of uniform smooth consistency, completely mixed, and shall not evolve gases.

3.5 Set Cement — After immersion in water for 5 days, the colour of any shade of the set cement, when viewed under water by natural daylight shall match the manufacturer's shade guide, if supplied, within the limits of professional acceptance.

3.6 Arsenic Content — The arsenic content of the material shall not be more than 2 ppm, when tested as prescribed in **A-3.**

3.7 Physical Properties — The material shall also comply with the requirements for physical properties given in Table 1 when tested according to prescribed methods.

TABLE 1 REQUIREMENTS OF ZINC PHOSPHATE DENTAL CEMENT FOR PHYSICAL PROPERTIES

Sl No.	Characteristic	Requirement		Method of Test (Ref to Cl No. of Appendix A)
		Class 1	Class 2	
(1)	(2)	(3)	(4)	(5)
i)	Net setting time* at 37°C, minutes	2·5 to 5·5	4·5 to 8·5	A-4
ii)	Compressive strength, MPa, *Min*	70	70	A-5
iii)	Film thickness, μm, *Max*	25†	25†	A-6
iv)	Water-leachable material, mgP_2O_5/g, *Max*	2·0	2	A-7

*The net setting time is determined from the completion of mixing, that is, total setting time less mixing time.

†The limit shall be 40 for Type 2 material.

3.8 Toxicity — The mixed cement, when used in accordance with the manufacturer's instructions, shall neither cause prolonged damage to oral tissues nor have any adverse systemic effect.

3.9 Manufacturer's Instructions — The manufacturer shall supply the instructions for bringing about physical contact of the powder and the liquid, and for the mixing of these materials to form the cement. The following details shall be included:

a) The temperature, condition and type of the slab and spatula;

b) The recommended powder/liquid ratio;

c) The rate of incorporation of the powder;

d) The time of mixing;

e) The maximum satisfactory working time after the end of mixing; and

f) A statement recommending that, when clinical conditions warrant, a liner should be placed between the cement and the dentine.

4. PACKING AND MARKING

4.1 Packing — The cement powder and liquid shall be supplied in properly sealed containers made of materials which shall not contaminate or permit contamination of the contents.

4.2 Instructions for Use — Instructions for proportioning the powder and liquid, and for manipulation of the cement shall accompany each package.

4.3 Marking — Each container shall bear legibly and indelibly the following information:

a) Name, type and class of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) The shade of the powder according to the manufacturer's shade guide, if supplied;

d) Net mass of the powder and the net volume of the liquid; and

e) Batch number.

4.3.1 The container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The method of preparing samples of the material and the criteria for conformity shall be as given in Appendix B or as agreed to between the purchaser and the supplier.

APPENDIX A

(*Clauses* 3.2.2, 3.4, 3.6 *and* Table 1)

METHODS OF TEST FOR ZINC PHOSPHATE DENTAL CEMENT

A-0. QUALITY OF REAGENTS

A-0.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Specification for water for general laboratory use (*second revision*).

A-1. PREPARATION OF TEST SPECIMENS

A-1.1 Ambient Conditions — Unless stated otherwise, the preparation of all specimens shall be carried out at 27 ± 2°C and at a relative humidity between 55 and 75 percent.

A-1.2 Components — All tests shall be carried out on specimens prepared from samples of the powder and liquid complying with **3.2** and **3.3**.

A-1.3 Apparatus for Mixing — All apparatus used for mixing and for testing shall be kept clean, dry and free from hardened particles of cement.

A-1.3.1 *Polished Glass Slab* — approximately 150 mm long, 75 mm wide, and 20 mm thick.

A-1.3.2 *Spatula* — inert to the cement.

A-1.4 Method of Mixing — Place known amounts of powder and liquid on the polished glass slab, and divide the powder into six separate portions as indicated in Table 2.

A-1.4.1 Using a linear, not rotary, motion of the spatula, with the edge sweeping approximately half the mixing area of the slab on each stroke, incorporate and mix the powder and liquid following the timing sequence indicated in Table 2. The total mixing time shall be 90 s and there shall be no particle of powder and no unused liquid remaining on the slab when mixing is completed.

TABLE 2 RATE OF INCORPORATION OF POWDER

Proportion of Total Amount of Powder	Time of Incorporation, Seconds
1/16	10
1/16	10
1/8	10
1/4	15
1/4	15
1/4	30

A-2. DETERMINATION OF POWDER/LIQUID RATIO FOR STANDARD TESTING CONSISTENCY

A-2.1 Apparatus

A-2.1.1 *Loading Device* — Of the type illustrated in Fig. 1, or an equivalent means whereby a force of 25 N (approx 2·5 kgf) may be applied vertically on to the cement. In Fig. 1, the anvil, which is attached to the bottom of the rod carrying the load, shall be horizontal

and parallel to the base and shall incorporate a device for holding the larger glass plate (*see* **A-2.1.2**) in contact with its surface. The second, smaller, glass plate shall be held on the base by guides to prevent movement or rotation when the load is applied. The load shall be capable of being applied smoothly and in such a manner that no rotational motion occurs. The two glass plates shall be capable of touching over their entire facing surfaces, without interference from guides, etc.

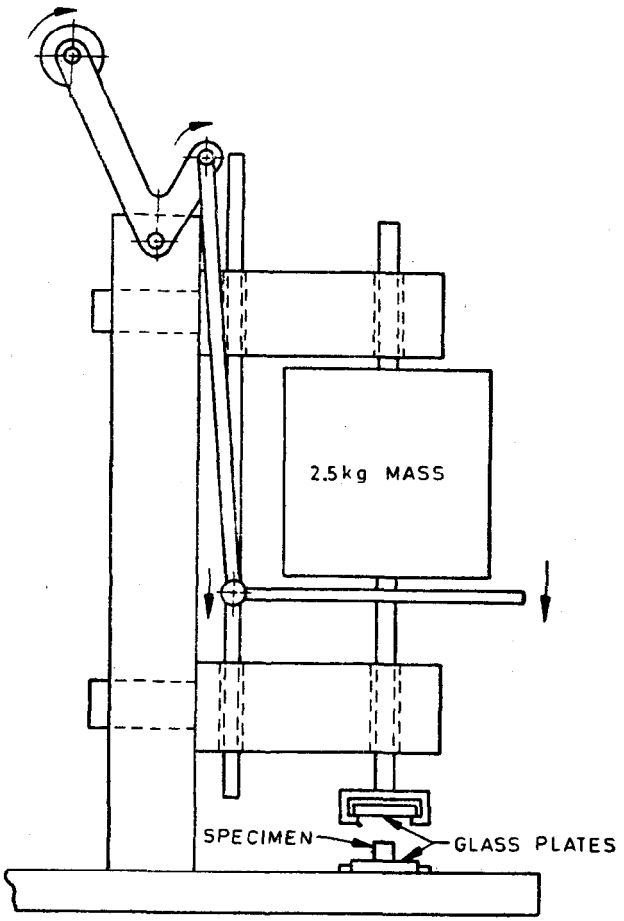

FIG. 1 LOADING DEVICE FOR MEASURING CONSISTENCY

A-2.1.2 *Two Flat Glass Plates* — approximately 50 and 40 mm square and approximately 5 mm thick.

A-2.1.3 *Measuring Device* — to deliver 0·075 ml of mixed cement in the form of a cylinder 6·0 mm high and 4·0 mm in diameter. A suitable device may consist of a glass tube and a PTFE plunger.

A-2.1.4 *Graduated Syringe Pipette* — having an accuracy of ± 0·001 ml.

A-2.2 Procedure

A-2.2.1 Carefully weigh out a trial amount of powder (200 to 300 mg) to an accuracy of 1 mg and transfer it to the glass mixing slab. Deliver 0·100 ml of liquid from the syringe pipette close to the powder.

A-2.2.2 After mixing in accordance with **A-1.4**, collect and load the cement into the measuring device. Deliver 0·075 ml of the mixed cement, preferably as an upright cylinder, onto the centre of the glass plate which is resting on the lower anvil of the loading device. If it is not possible to deliver all the cement from the measuring device in a single operation, take the residue with the tip of a clean spatula and place on the centre of the other glass plate. Position both glass plates relative to each other, without pressure, in such a way that any cement on the second glass plate contacts centrally the bulk of the cement on the first glass plate.

A-2.2.3 Sixty seconds after the completion of mixing, gently press the cement out between the two glass plates with a force of 25 N (approx 2·5 kgf), applied in a direction perpendicular to the lower glass plate.

A-2.2.4 After the cement has set, measure the major and minor diameters of the cement disc to an accuracy of 0·5 mm, and calculate the mean. If the two measurements differ by more than 1 mm, discard the result and repeat the test.

A-2.2.5 Make trial mixes of varying powder/liquid ratios until the mean diameter, calculated from the major and minor diameters measures, is 28 ± 1 mm. Check this result twice.

A-2.2.6 The powder/liquid ratio which gives the required mix consistency, called the 'standard testing consistency', shall be used in the preparation of all test specimens for tests carried out in accordance with this standard.

A-3. ARSENIC CONTENT

A-3.1 Preparation of Sample — Powder the set cement and sieve through a 75 μm (200 mesh) sieve. Disperse 2 g of the sieved powder in 30 ml of water and add 10 ml of hydrochloric acid, 38 percent (*m/m*) (d 1·19 g/ml). Use this solution in the test for total arsenic content.

A-3.2 Procedure — Determine the arsenic content of the material by any of the methods given in IS : 2088-1983*.

A-4. NET SETTING TIME

A-4.0 The setting time determined by this test method is measured from the completion of mixing, and not the more usual total setting time, where the time is measured from first contact between the cement components.

A-4.1 Apparatus

A-4.1.1 *Oven or Cabinet* — In which the specimen may be maintained at a temperature of 37 ± 1°C and a relative humidity of at least 30 percent.

A-4.1.2 *Indentor* — of mass 400 ± 5 g and having a flat end of diameter 1·0 ± 0·1 mm. The needle tip shall be cylindrical for a distance of approximately 5·0 mm. The needle end shall be plane and at right angles to the axis of the rod.

A-4.1.3 *Metal Mould* — Similar to that illustrated in Fig. 2.

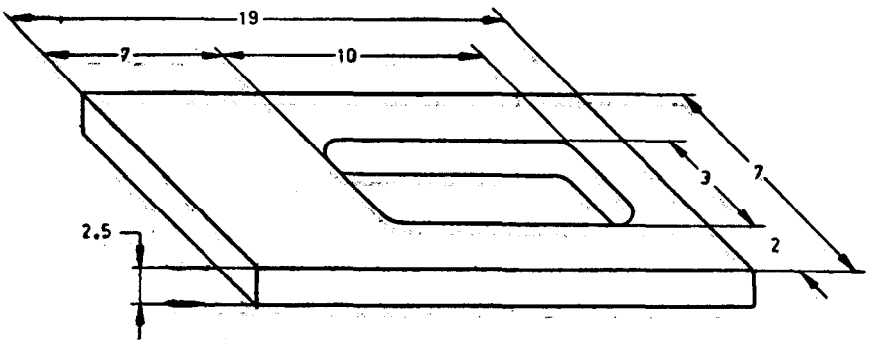

FIG. 2 MOULD FOR USE IN DETERMINING SETTING TIME

A-4.1.4 *Metal Block* — of minimum dimensions 8 × 20 × 10 mm, either as part of **A-4.1.1** or **A-4.1.2** or as a separate item.

A-4.1.5 *Soft Aluminium Foil*

A-4.2 Procedure

A-4.2.1 Place the metal rectangular mould, conditioned to 27 ± 2°C, on a piece of the aluminium foil of convenient size and fill to a level surface with cement.

*Methods for determination of arsenic (*second revision*).

A-4.2.2 One minute after the completion of mixing, place the assembly, comprising mould, foil and cement specimen, on the metal block, which has been conditioned to 37 ± 1°C, and replace in the oven. Ensure good contact between the mould, foil and metal block.

A-4.2.3 One and a half minutes after the completion of mixing, carefully lower the indentor vertically onto the surface of the cement and allow it to remain there for 5 seconds. Repeat this at intervals until near the expected time of setting, at which stage reduce the intervals to 15 seconds. Maintain the needle in a clean condition by cleaning, if necessary, between indentations.

A-4.2.4 Record the net setting time as the period of time which elapses from the completion of mixing to the time when the needle fails to make a perceptible circular indentation on the surface of the cement, when viewed under a hand lens of low magnification.

A-4.2.5 Take the mean of three such recorded results, rounded to the nearest 15 seconds, as the test result.

A-5. COMPRESSIVE STRENGTH

A-5.1 Apparatus

A-5.1.1 *Oven or Cabinet* — Maintained at a temperature of 37 ± 1°C and a relative humidity of at least 30 percent.

A-5.1.2 *Split Mould and Plates* — Such as those illustrated in Fig. 3, with internal dimensions 6 mm high and 4 mm diameter, and made of stainless steel or other suitable material that will not be attacked or corroded by the cement.

A-5.1.3 *Individual Screw Clamps*

A-5.1.4 *Compressive Strength Testing Apparatus* — With a cross-head speed of 0·75 ± 0·25 mm/min.

A-5.2 Preparation of Test Specimens

A-5.2.1 Bring the moulds, top and bottom plates and the screw clamps to 27 ± 2°C. Pack the cement, mixed to the standard testing consistency (*see* **A-2.2**), to a slight excess into the assembled split mould, within 1 min of the completion of mixing.

NOTE — In order to consolidate the cement and avoid trapping air, it is advisable to convey the largest convenient portions of mixed cement to the mould and apply to one side with a suitable instrument. Fill the mould to excess in this manner and then place on the bottom plate with some pressure.

A-5.2.2 Remove any bulk extruded cement, place the top metal plate in position and manually squeeze together. Put the mould and plates in the clamp and screw tightly together. Not later than 2 min after the completion of mixing, transfer the whole assembly to the oven maintained at 37 ± 1°C.

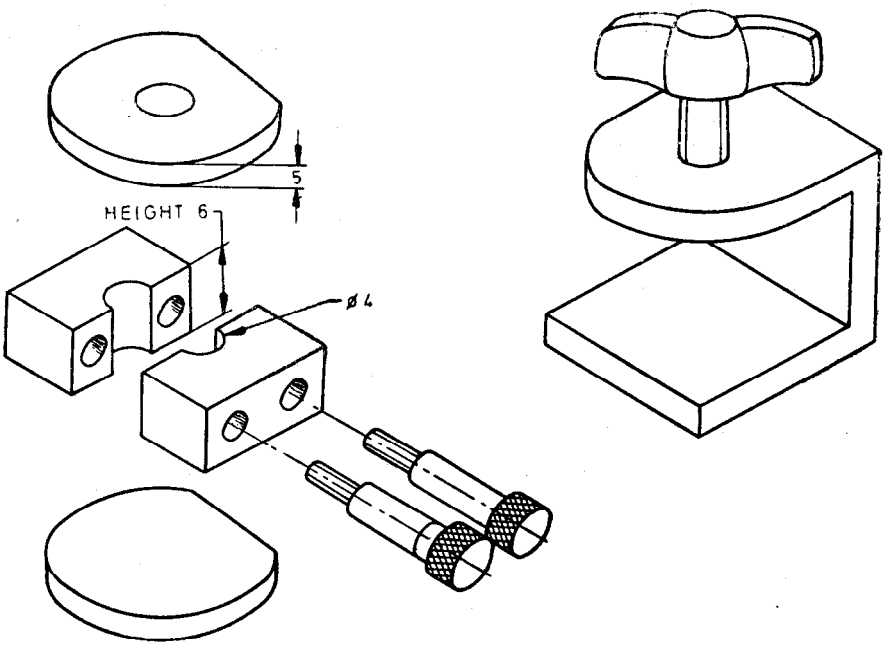

FIG. 3 MOULD AND CLAMP FOR PREPARATION OF COMPRESSION TEST SPECIMEN

A-5.2.3 One hour after the completion of mixing, remove the plates, and surface the ends of the specimen plane, at right angles to its long axis.

A-5.2.4 Grind the ends flat and remove any excess cement by drawing back and forth on a glass plate with a small amount of 350 mesh silicon carbide powder, maximum particle size 45 μm, mixed with water. Keep both ends of the specimen wet during the grinding and rotate about one quarter turn every few strokes.

A-5.2.5 Remove the specimen from the mould immediately after surfacing and check for air-voids or chipped edges. Discard any such defective specimens.

NOTE — To facilitate the removel of the hardened cement specimen, the internal surface of the mould may be evenly coated, prior to filling, with a 3 percent solution of micro-crystalline or paraffin wax in pure toluene. Alternatively a thin film of silicone grease or PTFE dry film lubricant may be used.

A-5.2.6 Immerse each acceptable specimen in distilled or deionized water and maintain at 37 $\pm$ 1°C for 23 h.

A-5.2.7 Five specimens shall be prepared and tested.

A-5.3 Procedure

A-5.3.1 Twenty-four hours after the completion of mixing, determine the compressive strength of the test specimens in the following manner, using a suitable apparatus with a cross-head speed of 0·75 ± 0·25 mm/min.

A-5.3.2 Place each specimen with the flat ends between the platens of the testing apparatus so that the load is applied in the long axis of the specimen.

A-5.3.3 Record the maximum load applied when the specimen fractures and calculate the compressive strength, in megapascals, using the formula.

A-5.4 Calculation

$$\text{Compressive strength, MPa} = \frac{4P}{\pi d^2}$$

where

P = maximum applied load, in newtons; and

d = diameter of the specimen, in millimetres.

If at least four of the five results obtained are below the limit specified in Table 2, the material shall be deemed to have failed the test. If at least four of the five results are above the limit specified in the table, the material shall be deemed to have passed the test. In other cases prepare a further 10 specimens and obtain the median result for all 15 specimens. Round this value to two significant figures and record as the compressive strength.

A-6. FILM THICKNESS

A-6.1 Apparatus

A-6.1.1 *Two Optically Flat Square or Circular Glass Plates* — Having a contact surface area of approximately 200 mm^2. Each plate shall be of a uniform thickness not less than 5 mm.

A-6.1.2 *Loading Device* — Of the type illustrated in Fig. 4, or an equivalent means whereby a force of 150 N (approx 15 kgf) may be applied vertically on to the cement. The bottom surface of the rod carrying the load shall be horizontal and parallel to the base and large enough to cover one of the glass plates. The load shall be capable of smooth application in such a manner that no rotational motion occurs. The glass plates shall be held on the base by guides to prevent movement or rotation when the load is applied.

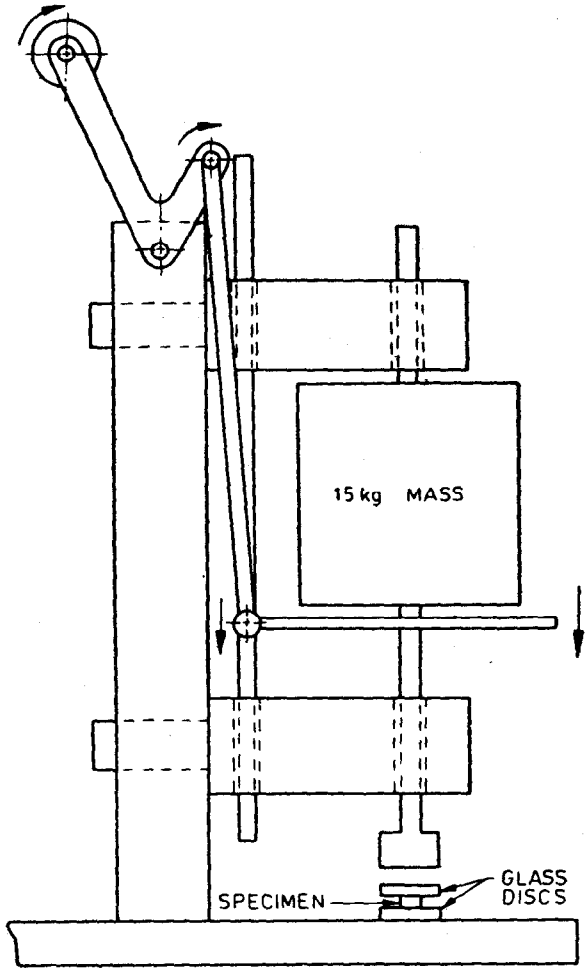

FIG. 4 LOADING DEVICE FOR FILM THICKNESS TEST

A-6.1.3 *Micrometer or Similar Measuring Instrument* — Accurate to 1 μm.

A-6.2 Procedure

A-6.2.1 Measure accurately the thickness of the two optically flat glass plates stacked in contact (reading A). Place a small quantity of cement, mixed to the standard testing consistency, on the centre of one of the glass plates and place the plate in the guides. Place the second glass plate centrally on the cement.

A-6.2.2 Three minutes after commencing the mix, carefully apply a force of 150 N (approx 15 kgf) vertically on the top plate and leave for 7 min. Ensure that the cement completely fills the area between the glass plates.

A-6.2.3 Ten minutes after the commencement of mixing, measure the thickness of the two glass plates and cement film (reading B).

A-6.2.4 The difference in thickness of the plates with and without the cement film (reading B — reading A) shall be taken as the thickness of the film. Report the mean result of three such tests to the nearest 5 μm.

A-7. WATER-LEACHABLE MATERIAL

A-7.1 Apparatus

A-7.1.1 *Oven or Cabinet* — Maintained at a temperature of 37 $\pm$ 1°C and a relative humidity at least 30 percent.

A-7.1.2 *Mould* — Consisting of a split brass or stainless steel ring contained in a former or retaining plate similar to that illustrated in Fig. 5. The height of the ring shall be 1·0 $\pm$ 0·003 mm and the internal diameter 10 mm. The former or retaining plate shall ensure that excess cement does not expand the split ring beyond a diameter of 10 mm.

A-7.1.3 *Individual Screw Clamps*

A-7.1.4 *Platinum Wire, Waxed Dental Floss* — or equivalent non-corrodible material.

A-7.1.5 *Two Wide-Mouthed Polyethylene Bottles* — of approximately 50 ml capacity, as illustrated in Fig. 6.

A-7.1.6 *Spectrophotometer* — Having a range including 650 nm, with cells (optional). Alternatively, a suitable comparator with Nessler tubes may be used.

A-7.2 Reagents

A-7.2.1 *Standard Phosphate Solution* — Dissolve 0·2 g of anhydrous disodium hydrogen orthophosphate (Na_2HPO_4) in one litre of water. This will give a solution containing the equivalent of 100 μg/ml of P_2O_5. Prepare a working standard containing 10·0 μg/ml of P_2O_5 by diluting 10 ml of this standard solution to 100 ml.

A-7.2.2 *Reagent I* — A 10 percent solution of ammonium molybdate in 1 N ammonia solution (33 ml of concentrated ammonia solution, 15 N in 500 ml of solution).

A-7.2.3 *Reagent II* — sulphuric acid, 20 N.

A-7.2.4 *Reagent III* — A 4-percent solution of ascorbic acid (it is essential that this solution be freshly prepared).

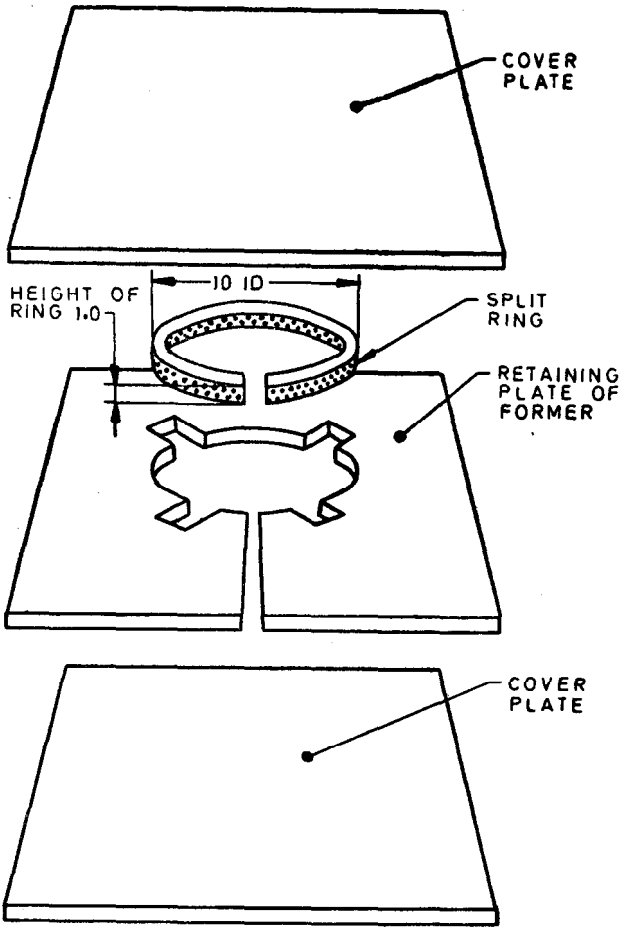

FIG. 5 MOULD FOR PREPARATION OF SPECIMEN FOR WATER LEACHABLE MATERIAL TEST

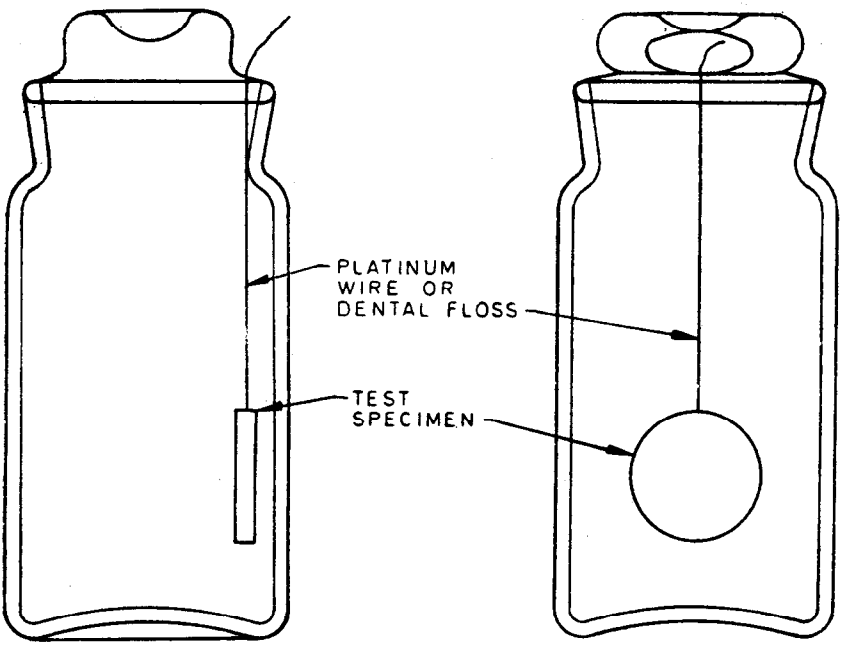

FIG. 6 WIDE-MOUTHED POLYETHYLENE BOTTLE CONTAINING SPECIMEN FOR WATER LEACHABLE MATERIAL TEST

A-7.2.5 *Reagent IV* — Mix 40 ml of reagent I and 60 ml of reagent II; allow to cool, and add 100 ml of reagent III. It is essential that this solution be freshly prepared.

A-7.3 Preparation of Test Specimen

A-7.3.1 Place the mould on a thin polyethylene or cellulose acetate sheetbacked by a flat plate.

A-7.3.2 Insert a convenient tared length of wire or dental floss through the split ring so that at least 4 mm projects into the ring. Fill the split ring with cement mixed to the standard testing consistency.

A-7.3.3 Cover with a further plate, faced with a sheet of polytheylene or cellulose acetate, press firmly together and apply the screw clamp.

A-7.3.4 Two minutes after the completion of mixing, place the mould, plates and the screw clamp into the oven maintained at 37 $\pm$ 1°C and a relative humidity of at least 30 percent.

A-7.3.5 After 1 hour, remove the plates and polyethylene or cellulose acetate sheets from the clamp and carefully separate the cement disc and attached wire or dental floss from the split ring. Remove any surplus cement from the edge of the disc and lightly brush the surface to remove any loose material.

A-7.4 Preparation of Test Solution — Weigh the specimen and immediately suspend it in 20 ml of water, contained in the polyethylene bottle, by means of the wire or dental floss. Ensure that the specimen does not touch the side of the bottle. Close the lid as tightly as possible and store for 23 hours at 37 ± 1°C.

A-7.5 Procedure

A-7.5.1 After 23 hours, remove the specimen from the water and determine, in duplicate, the amount of phosphate in solution by the following procedure.

A-7.5.2 Transfer the contents of each of the polyethylene bottles to a 50-ml flask and dilute to the calibration mark with water. Transfer 10 ml aliquot portions of these solutions to 50-ml volumetric flasks, and add 5 ml of reagent IV to each, the contents then being diluted to the calibration marks and thoroughly mixed. Treat 10 ml of the standard phosphate solution similarly by adding 5 ml of reagent IV and making the volume up to 50 ml in a volumetric flask. At the same time also prepare a blank. Allow these flasks to stand for 24 hours and then compare the solution at 650 nm in a suitable spectrophotometer. If no spectrophotometer is available the sample solution may be compared against a suitable standard; 8 ml of the working standard solution (**A-7.2.1**) approximates to the specification limit if the cement disc is of 0·2 g mass. Standard Nessler procedures should be adopted, but where any result is questionable, the spectrophotometer method shall be used.

A-7.6 Expression of Results — The amount of water-leachable material, expressed as P_2O_5 eluted in milligrams per gram of the specimen, is given by the formula:

$$\frac{A_1 - A_3}{A_2 - A_3} \times \frac{1}{2\,m}$$

where

A_1 = absorbance of the test solution;

A_2 = absorbance of the standard phosphate solution;

A_3 = absorbance of the blank solution; and

m = mass, in grams, of the specimen.

NOTE — The absorbance, A_2, of the standard phosphate solution measured in a 1-cm cell at 650 nm is normally about 0·260.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF ZINC PHOSPHATE CEMENT

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the precautions and directions given in **B-1.1** to **B-1.7** shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry, air-tight glass or other suitable containers.

B-1.6 The sample containers shall be of such size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material of the same shade, same type, and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of this specification.

B-2.2 The number of containers (n) to be selected from the lot shall depend on the size of the lot (N) and shall be as given in Table 2, subject to the provision that if n containers do not provide sufficient material for carrying out all the tests specified in **3**, then at least as many containers as will provide sufficient material shall be taken out.

TABLE 3 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

Lot Size N	Number of Containers to be Selected n
(1)	(2)
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 to 1 000	7

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and in order to ensure the randomness of selection the random sampling method given in IS : 4905-1968* may be followed.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 *Liquid Component* — Empty the contents of all the sample containers selected into a clean glass-stoppered bottle. Thoroughly mix the contents and divide the composite sample into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-3.1.2 *Solid Component* — Empty the contents of all the sample containers selected into a square-sided jar having a capacity of 2 litres and a self-sealing cap. Rotate the jar on its minor axis for two hours at the rate of 25 rev/min. Divide the composite sample into three equal parts, one for the purchaser, another for the supplier and third for the referee.

B-3.2 Referee Sample — The referee sample shall consist of one composite sample each of the solid component and the liquid component, marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute.

B-4. NUMBER OF TESTS

B-4.1 Tests for the requirements given in **3.2** and **3.3** shall be tested visually on the composite sample of solid and liquid component respectively. If the sample conforms to the above requirement, the tests for all the characteristics given in **3** and Table 1 shall be conducted on the composite sample made out of liquid and solid components.

*Methods for random sampling.

B-5. CRITERIA FOR CONFORMITY

B-5.1 A lot shall be declared as conforming to this specification if the composite sample satisfies the requirements for each of the characteristics listed in 3 and in Table 1. If the requirements for any of the characteristics are not met, the lot shall be declared to have not satisfied the requirements of the specification.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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